

Improved High Mass Range Resolution with an Omegatron Mass Spectrometer

J. H. BLOOM C. E. LUDINGTON R. L. PHIPPS

MAY 3 1963

Requests for additional copies by Agencies of the Department of Defense, their contractors, and other government agencies should be directed to the:

Armed Services Technical Information Agency Arlington Hall Station Arlington 12, Virginia

Department of Defense contractors must be established for ASTIA services, or have their 'need-to-know' certified by the cognizant military agency of their project or contract.

All other persons and organizations should apply to the:

U. S. DEPARTMENT OF COMMERCE OFFICE OF TECHNICAL SERVICES, WASHINGTON 25, D. C.

APCRL-62-953 OCTOBER 1962



Improved High Mass Range Resolution with an Omegatron Mass Spectrometer

J. H. BLOOM C. E. LUDINGTON R. L. PHIPPS

Abstract

The influence of changing the magnetic field on the resolution and sensitivity of the omegatron mass spectrometer is shown for krypton. Good resolution may be extended through the mass range of the xenon isotopes (mass number 136) by carefully selecting the operating parameters of the omegatron. The resolution is plotted against the magnetic field strength for Kr⁸⁴⁺, and follows the theoretical predictions within experimental error. The effect of varying the other parameters of the omegatron with krypton is shown and discussed. A summation of the work with xenon is also given.

Acknowledgment

The authors wish to express sincere appreciation to Miss Olive N. L. Denniston and Mr. Lawrence C. Mansur for their critical review of this paper.

Contents

		Page
1.	INTRODUCTION	1
2.	EXPERIMENTAL SYSTEM	3
3.	PROCEDURE	4
4.	CONCLUSION	9
	References	11
	Bibliography	11

Improved High Mass Range Resolution with an Omegatron Mass Spectrometer*

1. INTRODUCTION

The need for identifying residual gases occupies a place of first importance in the advancement of ultra-high vacuum research. A low pressure measurement alone does not assure gas ambient purity. At least one harmful constituent may still remain. Identification of residual gases is essential to the study of many phenomena in vacuum with a sensitivity to surface contamination.

The omegatron mass spectrometer is a practical tool in fulfilling this need. Unfortunately, much of the literature states that the instrument is useful only through mass number 44. One notable exception to this purported limitation will be found in the works of Dummler, who reports good resolution through mass number 100.

The work performed in our laboratory extends the range of the omegatron still further. To date, good resolution is achieved through mass number 136. The omegatron clearly resolves seven of the xenon isotopes in the correct percentages of natural abundance, within experimental error.

The magnetic field parameter had the greatest influence on the resolution of the omegatron in the high mass range. An increase in the magnetic field re-

^{*}Presented at the Sixth National Conference on Tube Techniques, New York City, September, 1962, Advisory Group on Electron Devices, Office of the Director of Defense Research and Engineering.

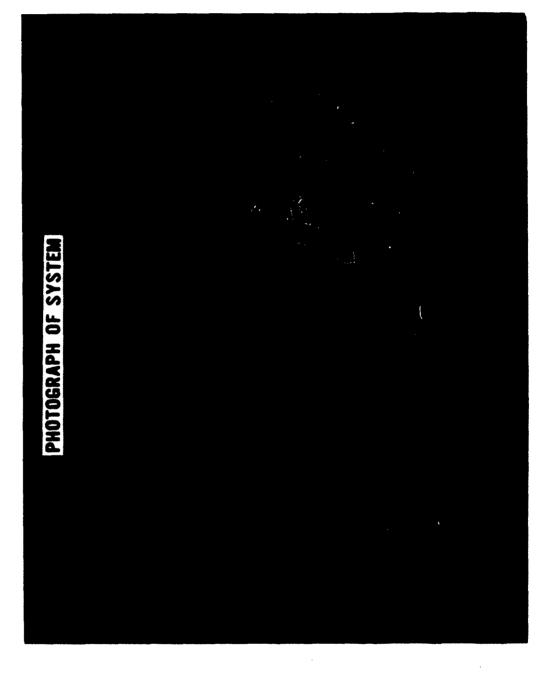


Figure 1. Photograph of System

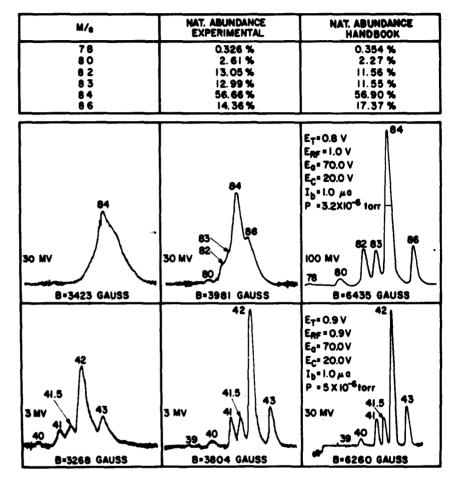


Figure 2. Variation of Resolution with Magnetic Field

multiple isotopes in the high mass region.

Magnetic field had a pronounced effect on the resolution, as can be seen in Figure 2. Note the spectra of krypton at different magnetic field strengths, as illustrated. The upper portion of the figure shows almost complete separation of the singly-ionized peaks at 6435 gauss. Based on later studies with xenon in which complete separation of the isotopes was obtained by increased precision in setting the trapping and r-f voltages, it is evident that similar results would have been achieved with krypton, if time had permitted. The percentages of the natural abundance of the mass peaks were approximately in the correct propor-

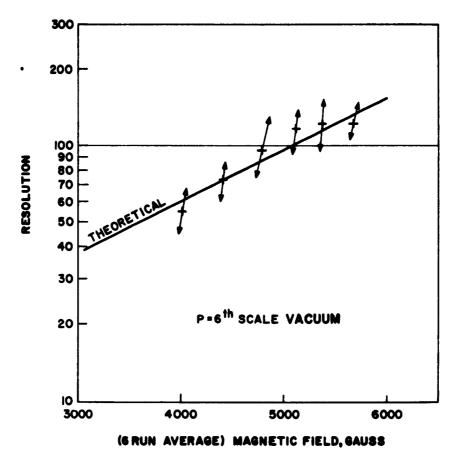


Figure 3. Average Resolution vs. Average Magnetic Field for Kr 84⁺

tion. Both experimental and handbook values of the natural abundance of the krypton isotopes are listed in the table of Figure 2. With the experimental data agreeing so closely with the handbook values, it is apparent that the omegatron is suitable for more than rough measurements of residual gases. The lower portion of Figure 2 shows a similar relationship for the doubly-ionized krypton isotopes. Complete separation between each mass peak was observed at 6260 gauss.

Figure 3 presents a plot of the resolution versus the magnetic field, with each plotted point representing an average of six runs. Arrows designate the range of the data. The straight line is a square-law plot (that is, R versus B^2),

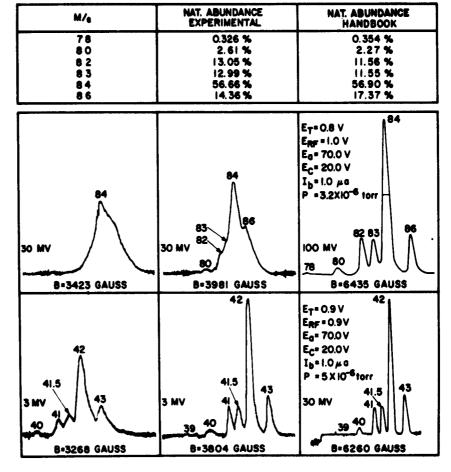


Figure 2. Variation of Resolution with Magnetic Field

multiple isotopes in the high mass region.

Magnetic field had a pronounced effect on the resolution, as can be seen in Figure 2. Note the spectra of krypton at different magnetic field strengths, as illustrated. The upper portion of the figure shows almost complete separation of the singly-ionized peaks at 6435 gauss. Based on later studies with xenon in which complete separation of the isotopes was obtained by increased precision in setting the trapping and r-f voltages, it is evident that similar results would have been achieved with krypton, if time had permitted. The percentages of the natural abundance of the mass peaks were approximately in the correct propor-

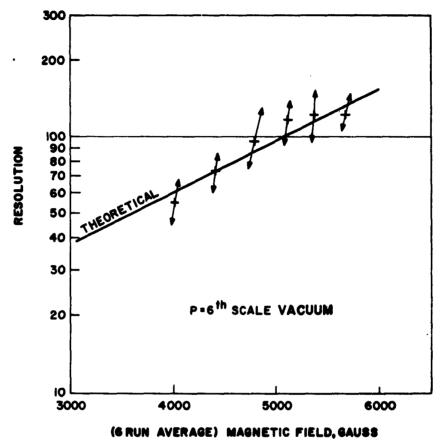


Figure 3. Average Resolution vs. Average Magnetic Field for Kr 84^{+}

tion. Both experimental and handbook values of the natural abundance of the krypton isotopes are listed in the table of Figure 2. With the experimental data agreeing so closely with the handbook values, it is apparent that the omegatron is suitable for more than rough measurements of residual gases. The lower portion of Figure 2 shows a similar relationship for the doubly-ionized krypton isotopes. Complete separation between each mass peak was observed at 8260 gauss.

Figure 3 presents a plot of the resolution versus the magnetic field, with each plotted point representing an average of six runs. Arrows designate the range of the data. The straight line is a square-law plot (that is, R versus B²).

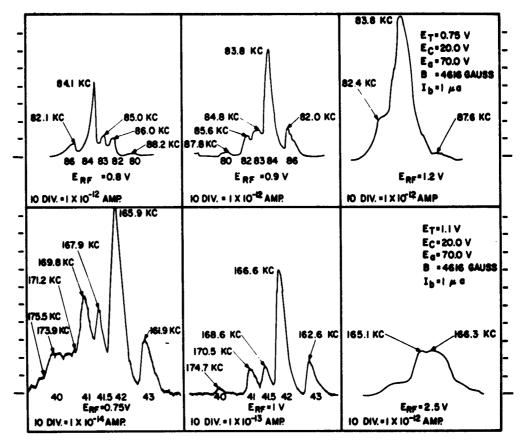


Figure 4. Variation of Resolution with r-f Voltage

The experimental points agree within the estimated error.

The importance of precise changes in the r-f and trapping voltages as scans are made in the high mass region is evident in Figures 4 and 5. Figure 4 shows some spectra of the krypton isotopes at various r-f voltages. The optimum setting for the singly-ionized krypton isotopes was 0.8 volt, while 1 volt seemed to be the best r-f voltage setting for the doubly-ionized isotopes. One can readily see that the identity of the isotopes is lost if the setting is either too high or too low.

Figure 5 is a series of plotted curves showing ion current versus trapping voltage for krypton. The optimum trapping voltage for Kr^{\dagger} was between 0.75 and 0.8 volts, and between 0.85 and 0.9 volts for $Kr^{\dagger \dagger}$.

Figure 6 shows a series of curves of the ion current versus the accelerating

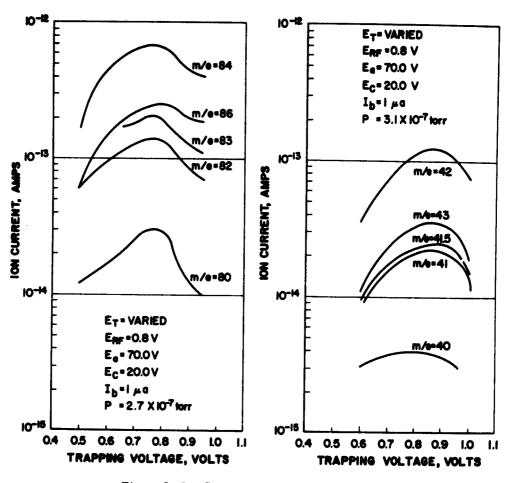


Figure 5. Ion Current vs. Trapping Voltage for Kr

voltage. The ion current was maximum between 60 and 70 volts. This corresponds to the point of maximum ionization efficiency for krypton. The appearance potential can also be estimated from this curve.

With the knowledge gained from the study of the omegatron with krypton, the experiment was extended to include some tests with xenon. Complete separation of seven of the xenon isotopes was obtained by increasing the magnetic field to 6486 gauss and tuning the r-f and trapping voltages one against the other. A very slight change in either the trapping or the r-f voltage had a marked effect on the xenon pattern. Figure 7 shows a spectrum of the xenon isotopes at the

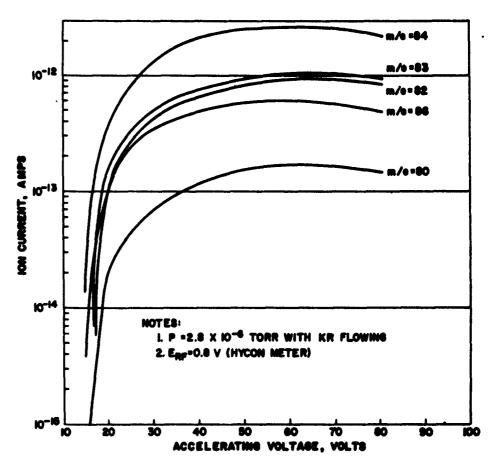


Figure 6. Ion Current vs. Accelerating Voltage

best settings. The table included in Figure 7 shows that the percentages of natural abundance were approximately correct.

4. CONCLUSION

The range of an omegatron may be extended through mass number 136 with good resolution by increasing the magnetic field and decreasing the r-f and trapping voltages. The settings of the r-f and trapping voltages are very

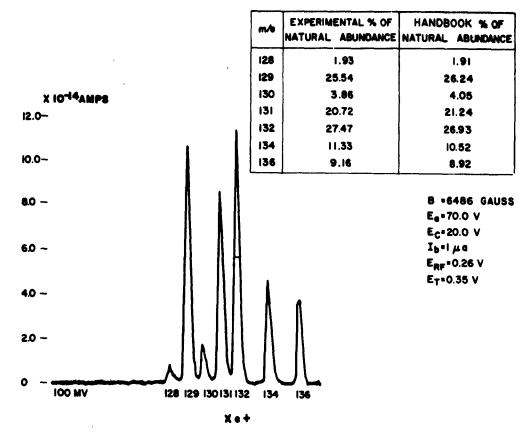


Figure 7. Xenon Spectrum

critical; they should be tuned one against the other. Time did not permit these later findings with xenon to be repeated with krypton. If the r-f and trapping voltages were tuned as precisely for the krypton tests as for the xenon tests, complete separation of the krypton isotopes would have been achieved.

This experiment also shows that R_N is proportional to B^2 . The omegatron has proved to be a practical tool for the analysis of partial pressures through mass number 136. Continued studies, undoubtedly, will extend this range even further.

References

- S. Dummler, "The Use of the Omegatron for Quantitative Partial Pressure Analysis in High Vacuum," Parts I and II, Vakuum-Technik, December, 1960, 131, 184.
- W. R. Watson, R. A. Wallace, and J. Lech, "Operational Data on the Omegatron as a Vacuum Analyzer," Vacuum Symposium Transactions, 1960, 421.

Bibliography

Alpert, D., and Buritz, R. S., "Ultra-High Vacuum, II. Limiting Factors on the Attainment of Very Low Pressure" (Westinghouse Research Labs., East Pitts-burgh, Pa. [Journal of Applied Physics, XXV, February, 1953]), 202.

Baker, F. A., and Giorgi, T. A., "Sorption and Descrption of Gas in a Hot-Cathode Ionization Gauge" (The Polytechnic, London: S.A.E.S. Getters, Milan, Italy [British Journal of Applied Physics, II, September 1960]), 433.

Berry, C. E., "Ion Trajectories in the Omegatron" (Consolidated Engineering Corp., Pasadena, Calif. [Journal of Applied Physics, XXV, January, 1953]), 28.

Dummler, S., "The Use of the Omegatron for Quantitative Partial Pressure Analysis in High Vacuum" (Institute of Applied Physics, Mainz University, Germany [Vakuum-Technik, Part I, V; Part II, VI, December, 1960]), 131, 184.

Edwards, A. G., "Some Properties of a Simple Omegatron-Type Mass Spectrometer" (Research Labs. Associated, Electrical Industries Ltd., Aldermaston, Berks. [British Journal of Applied Physics, September, 1954], 44.

- Hebling, A., and Lichtman, D., "Fragment Patterns and Appearance Potentials Using the Omegatron Mass Spectrometer" (Sperry Gyroscope Co., Great Neck, N. Y. [A.S.T.M. Ninth Annual Conf. on Mass Spectrometry, Chicago, Ill., June, 1961]).
- Klopfer, A., and Schmidt, W., "An Omegatron Mass Spectrometer and Its Characteristics," Vacuum, X (November 1960), 363.
- Kornelsen, E. V., "An Ultra-High Vacuum Mass Spectrometer" (National Research Council, Ottawa, Canada [19th Physical Electronics Conference, M.I.T., 1959]), 156.
- Lafferty, J. M., and Vanderslice, T. A., "The Interplay of Electronics and Vacuum Technology" (General Electric Research Lab., Schenectady, N. Y., Report No. 61-RL-2615G).
- Levine, L., and Lichtman, D., "Analysis of Gas Evolution from a Titanium Hydride Gas Generator" (Sperry Gyroscope Co., Great Neck, N. Y. [Review of Scientific Instruments, XXXI, July, 1960]), 731.
- Lichtman, D., "Methane Evolution in Ion Pump Systems and Water Vapor Reaction in an Omegatron Structure" (Sperry Gyroscope Co., Great Neck, N. Y. [Am. Phys. Soc. Annual Meeting, New York, January, 1960; Phys. Elec. Conference, March, 1960, Cambridge, Mass.]).
- Lichtman, D., "Use of the Omegatron in the Determination of Parameters Affecting Limiting Pressures in Vacuum Devices" (Sperry Gyroscope Co., Great Neck, N. Y. [Journal of Applied Physics, XXXI, July, 1960]), 1213.
- Lichtman, D., and Hebling, A., "Ultra-High Vacuum Demountable System for Mass Spectrometer Gas Analysis" (Sperry Gyroscope Co., Great Neck, N. Y. [Seventh National Symposium, Cleveland, Ohio, October 12-14, 1960; Vacuum Symposium Transactions, 1960]), 187.
- Margoninski, Y., Wolsky, S. P., and Zdanuk, E., "Properties of an Omegatron Tube Employing a Lanthanum Boride Filament" (Raytheon Co., Waltham, Mass., Technical Memorandum, June, 1961), 1.
- Morrison, J. D., "Studies of Ionization Efficiency, I. The Determination of Molecular Appearance Potentials Using the Mass Spectrometer" (Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia [Journal of Chemical Physics, XIX, October, 1951]), 1305.
- Nicholson, A. J. C., "Measurement of Ionization Potentials by Electron Impact" (Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia (Journal of Chemical Physics, XXIX, December, 1958)), 1312.
- Reich, G., "Sensitivity and Resolving Power of the Omegatron" (Koln. Hochvakuum-Entwicklungsabteilung [Script of lecture given at Conference of a Mass Spectroscopy work team, April 20, 1961, Bad Nauheim; Vakuum-Technik VI]), 242.
- Roehrig, J. R., "Development of Satellite Instrumentation Reference Standards and Calibration Equipment for Measurements at Very Low Gas Densities" (National Research Corporation, Cambridge, Mass., Technical Report No. 2, National Aeronautics and Space Adm. Contract NAS5-14, January, 1960).
- Schlier, R. E., "Adsorption of Oxygen and Carbon Monoxide in Tungsten" (Barus Res. Lab., Brown Univ. [Journal of Applied Physics, XXIX, August, 1958]), 1162.

References

- S. Dummler, "The Use of the Omegatron for Quantitative Partial Pressure Analysis in High Vacuum," Parts I and II, <u>Vakuum-Technik</u>, December, 1960, 131, 184.
- 2. W. R. Watson, R. A. Wallace, and J. Lech, "Operational Data on the Omegatron as a Vacuum Analyzer," Vacuum Symposium Transactions, 1960, 421.

Bibliography

Alpert, D., and Buritz, R. S., "Ultra-High Vacuum, H. Limiting Factors on the Attainment of Very Low Pressure" (Westinghouse Research Labs., East Pitts-burgh, Pa. (Journal of Applied Physics, XXV, February, 1953)), 202.

Baker, F. A., and Giorgi, T. A., "Sorption and Desorption of Gas in a Hot-Cathode Ionization Gauge" (The Polytechnic, London: S.A.E.S. Getters, Milan, Italy [British Journal of Applied Physics, II, September 1960]), 433.

Berry, C. E., "Ion Trajectories in the Omegatron" (Consolidated Engineering Corp., Pasadena, Calif. [Journal of Applied Physics, XXV, January, 1953]), 28.

Dummler, S., "The Use of the Omegatron for Quantitative Partial Pressure Analysis in High Vacuum" (Institute of Applied Physics, Mainz University, Germany [Vakuum-Technik, Part I, V; Part II, VI, December, 1960]), 131, 184.

Edwards, A. G., "Some Properties of a Simple Omegatron-Type Mass Spectrometer" (Research Labs. Associated, Electrical Industries Ltd., Aldermaston, Berks. [British Journal of Applied Physics, September, 1954], 44.

Hebling, A., and Lichtman, D., "Fragment Patterns and Appearance Potentials Using the Omegatron Mass Spectrometer" (Sperry Gyroscope Co., Great Neck, N. Y. [A.S.T.M. Ninth Annual Conf. on Mass Spectrometry, Chicago, Ill., June, 1961]).

Klopfer, A., and Schmidt, W., "An Omegatron Mass Spectrometer and Its Characteristics," <u>Vacuum</u>, X (November 1960), 363.

Kornelsen, E. V., "An Ultra-High Vacuum Mass Spectrometer" (National Research Council, Ottawa, Canada [19th Physical Electronics Conference, M.I.T., 1959]), 156.

Lafferty, J. M., and Vanderslice, T. A., "The Interplay of Electronics and Vacuum Technology" (General Electric Research Lab., Schenectady, N. Y., Report No. 61-RL-2615G).

Levine, L., and Lichtman, D., "Analysis of Gas Evolution from a Titanium Hydride Gas Generator" (Sperry Gyroscope Co., Great Neck, N. Y. [Review of Scientific Instruments, XXXI, July, 1960]), 731.

Lichtman, D., "Methane Evolution in Ion Pump Systems and Water Vapor Reaction in an Omegatron Structure" (Sperry Gyroscope Co., Great Neck, N. Y. [Am. Phys. Soc. Annual Meeting, New York, January, 1960; Phys. Elec. Conference, March, 1960, Cambridge, Mass.]).

Lichtman, D., "Use of the Omegatron in the Determination of Parameters Affecting Limiting Pressures in Vacuum Devices" (Sperry Gyroscope Co., Great Neck, N. Y. [Journal of Applied Physics, XXXI, July, 1960]), 1213.

Lichtman, D., and Hebling, A., "Ultra-High Vacuum Demountable System for Mass Spectrometer Gas Analysis" (Sperry Gyroscope Co., Great Neck, N. Y. [Seventh National Symposium, Cleveland, Ohio, October 12-14, 1960; Vacuum Symposium Transactions, 1960]), 187.

Margoninski, Y., Wolsky, S. P., and Zdanuk, E., "Properties of an Omegatron Tube Employing a Lanthanum Boride Filament" (Raytheon Co., Waltham, Mass., Technical Memorandum, June, 1961), 1.

Morrison, J. D., "Studies of Ionization Efficiency, I. The Determination of Molecular Appearance Potentials Using the Mass Spectrometer" (Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia [Journal of Chemical Physics, XIX, October, 1951]), 1305.

Nicholson, A. J. C., "Measurement of Ionization Potentials by Electron Impact" (Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia [Journal of Chemical Physics, XXIX, December, 1958]), 1312.

Reich, G., "Sensitivity and Resolving Power of the Omegatron" (Koln. Hochvakuum-Entwicklungsabteilung [Script of lecture given at Conference of a Mass Spectroscopy work team, April 20, 1961, Bad Nauheim; Vakuum-Technik VI]), 242.

Roehrig, J. R., "Development of Satellite Instrumentation Reference Standards and Calibration Equipment for Measurements at Very Low Gas Densities" (National Research Corporation, Cambridge, Mass., Technical Report No. 2, National Aeronautics and Space Adm. Contract NAS5-14, January, 1960).

Schlier, R. E., "Adsorption of Oxygen and Carbon Monoxide in Tungsten" (Barus Res. Lab., Brown Univ. [Journal of Applied Physics, XXIX, August, 1958]), 1162.

Appendix A

Figure A-1 is a block diagram showing the functional relationship of the components of the system. A few comments about some of the components follow.

A mechanical forepump, block 1, is used for the initial pumpdown of the system. A clean copper-foil trap, block 2, placed between the pump and the system eliminates the risk of oil contamination. These components are sealed off once the Vac-Ion pump, block 3, starts.

The laboratory-built electromagnet, block 8, is capable of producing a variable magnetic field up to 7000 gauss. The magnet slides in and out of the oven on an adjustable mount.

The omegatron power supply,* block 9, also built in the laboratory, is a modified Raytheon design. The circuit diagram is shown in Figure A-2.

A strip chart and an X-Y recorder, block 13, are used for recording the test data. Advantages can be cited for each.

V1, V2, V3 and V4 are Granville-Phillips Type C bakeable metal valves.

The system is pumped on continuously to prevent build-up of any contaminants that may be present. The continuous flow of gas is controlled by a Granville-Phillips Type C valve.

^{*}Design modified by Ernest Owen.

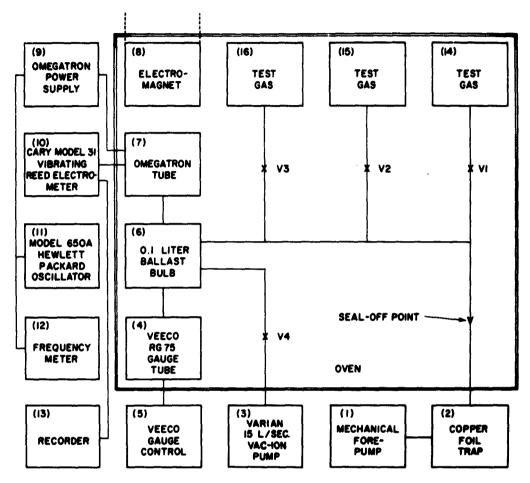


Figure A-1. A Block Diagram of Experimental Setup

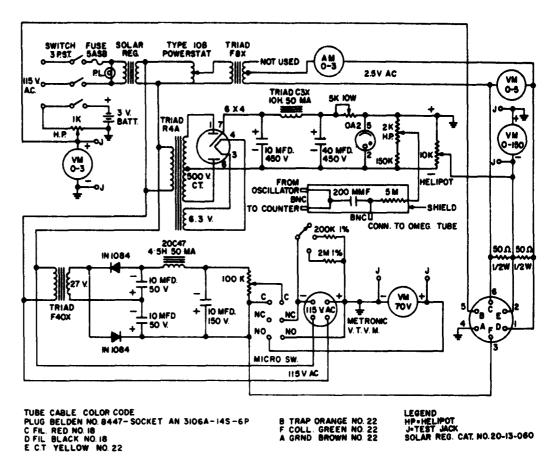


Figure A-2. Omegatron Power Supply

UNCLASSIFIED Residual gas Mass spectrum analysis Bloom, J. H. Ludington, C. E. Phipps, R. L.	UNCLASSIFIED	UNCLASSIFIED Residual gas Mass spectrum analysis Bloom, J. H. Ludington, C. E. Phipps, R. L.
1111	 	
AF Cambridge Research Laboratories, Badford, Mass. Electronics Research Directorate IMPROVED HIGH MASS RANGE RESCUTION WITH AN OFFICATION WITH AN OFFICATION HASS SPECTHOMETE, by J. H. Bloom, C. E. Ludington, and R. L. Phipps. 21 p. Incl. illus. October 1962 Uncleasified report AFCRI-62-953 Uncleasified report The influence of changing the magnetic field on the resolution and sensitivity of the owagatron mass apectrometer is shown for krypton. Good resolution may be extended through the mass range of the xenon isotopes (mass number 136) by carefully selecting the operating parameters of the omagatron. The resolution is plottagl, against the magnetic field strength for Kr of the omagatron. The resolution is plottagl, and follows the theoretical predictions within experimental error. The effect of varying the other parameters of the omagatron with krypton is shown and discussed. A summation of the		AF Cambridge Research Laboratories, Bedford, Hass. Electronics Research Directorate IMPROVED HIGH MASS RANGE RESOLUTION WITH AN OFFICATION MASS SPECTROMETER, by J. H. Bloom. C. E. Ludington, and R. L. Phipps. 21 p. incl. illus. October 1962 Unclassified report AFCRI-62-953 Unclassified report The influence of changing the magnetic field on the resolution and sensitivity of the omegatron mass spectrometer is shown for krypton. Good resolution may be extended through the mass range of the xenon isotopes (mass number 136) by carefully selecting the operating parameters of the omegatron. The resolution is plotting, against the magnetic field strength for Kryfton experimental error. The effect of varying the other parameters of the omegatron with krypton is shown and discussed. A summation of the work with xenon is also given.
UNCLASSIFIED 1. Residual ges 2. Mass spectrus analysis 1. Bloom, J. H. II. Ludington, C. E. III. Phipps, R. L.	UNCLASSIFIED	UNCLASSIFIED 1. Residual gas 2. Mass spectrum analysis 1. Bloom, J. H. II. Ludington, C. E. III. Phipps, R. L.
AF Cambridge Research Laboratories, Bedford, Hass. Electronics Research Directorate EMENDVED HIGH MASS SPACE BESOLUTION WITH AN ORDIGATION MASS SPECTMONETR, by J. H. Bloom, C. E. Ludiagron, and R. L. Phipps. 21 p. incl. 111us. October 1962. Unclassified report The influence of changing the magnetic field on the resolution and sensitivity of the comparence resolution and sensitivity of the comparence resolution may be extended through the mass renge of the xenon isotopes (mass number 136) by carrelly selecting the operating parameters of the comparence. The resolution is plotting against the magnetic field strength for Kr and follows the theoretical predictions within emperimental strow. The effect of varying the other parameters of the comparison within is shown and discussed. A summation of the work with menon is also given.		AF Cambridge Research Laboratories, Bedford, Ness. Electronics Research Directorate INTERPORD HIGH MASS TANCE RESOLUTION WITH AN GENERATION HASS SPECTROMETER, by J. H. Bloom, C. E. Ladington, and R. L. Phipps. 21 p. incl. 11lus. October 1962 Unclassified report The influence of changing the magnetic field on the resolution and sensitivity of the omegatron the resolution and sensitivity of the omegatron tessulation may be axtended through the mass range of the amon isotopes (mass number 136) by carefully selecting the operating parameters of the campairon. The resolution is plotting and fallows the theoretical predictions within experimental error. The effect of varying the ether parameters of the omegatron with krypton te shown and discussed. A summation of the work with memon is also given.